

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Rahul K. Dharmadhikary                      Examiner: Lynda Salvatore  
Serial No.: 10/535,498                                      Art Unit: 1771  
Filed: 05/18/2005  
For: ETHYLENE OXIDE STERILIZABLE, LOW-COST NONWOVEN  
LAMINATES WITH HIGH WET PEEL STRENGTH AND IMPROVED  
BARRIER PROPERTIES

Commissioner for Patents  
P. O. Box 1450  
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**DECLARATION UNDER 37 C.F.R. 1.132**

Rahul K. Dharmadhikary declares as follows:

1. I am one of the inventors of the subject matter of the above-identified patent application.
2. I received a Bachelor of Science degree in Textiles in the year 1989 and a Master of Science degree in Textiles in the year 1991 from the University of Maryland located at College Park, MD. In the year 1995, I was awarded the degree of Doctor of Philosophy in Fiber and Polymer Science from North Carolina State University located at Raleigh, NC.
3. From April 1998 to date, I have been employed by Ahlstrom Windsor Locks, LLC, the assignee of the above-identified application. My present position is Director of Technology, Medical Fabrics.
4. Under my direction and control, non-woven laminates were prepared under conditions as set forth here below:

**Example 1:**

A three layer (A-B-A) co-extruded film was prepared. Core layer (B) was made from a LDPE (PE 1017). This linear low density polyethylene is commercially available from Chevron Philips Chemical Company, The Woodlands, Texas. The properties of PE 1017 resin (MSDS No. PL001) are a melt index of 7.0 gms/10 min (ASTM D1238-57T) and a density of 0.917 gms/cc (ASTM D1505-60T). It is characterized by excellent processability, high temperature stability and low neck-in. The color is translucent to clear.

The outer layers (A) were made using MARLEX EX01D resin, which is commercially available from Philips Sumika Polypropylene Company (now Chevron Philips Chemical Company, The Woodlands, Texas). MARLEX EX01D is a blend of polypropylene and polyethylene (approximately 85% polypropylene and 15% polyethylene). Its properties are density – 0.906 g/cc (ASTM D1505); melt flow (230 °C) – 35 g/10 min (ASTM D1238); melt point – 167°C (ASTM D3418); and crystallization temperature – 110 °C (ASTM D3418). To produce the co-extruded film, Marlex EX 01 D resin was fed from an extruder A while LDPE 1017 was fed from an extruder B. The two resins were then fed to a three layer feed-block and an ABA selector plug to produce the ABA co-ex film. Extruded ratio used in this particular example was 10-80-10 by weight.

The equipment required to produce co-extruded films is available from Cloeren Incorporated, Texas, USA. Extruder A is a 2.5” size, 5 zone extruder with a temperature profile from about 400 °F in the 1<sup>st</sup> zone increasing to approximately

about 555 °F in the 5<sup>th</sup> zone. Extruder B is a 3.5” size, 6 zone extruder with a temperature profile of about 400 °F in the 1<sup>st</sup> zone increasing to about 555 °F in the 6<sup>th</sup> zone. The die is maintained at approximately 555 °F.

Each side of the cast film was laminated to a PP spunbond substrate using the hot melt adhesive Fuller NW 1023, which is commercially available from H.B. Fuller Company, St. Paul, Minnesota. The adhesive is applied in a fiberized spray pattern at a processing temperature of between 275 and 300 °F. Line speed for the lamination process was about 300 fpm. This hot melt adhesive has the following physical properties: Molten Gardner color – 3; Mettler Soft Point (ASTM D3461) – 200 °F; specific gravity – 0.96; viscosity at 275 °F – 11, 500 cP; viscosity at 300 °F – 3,235 cP; viscosity at 325 °F – 1,925 cP and viscosity at 350 °F – 1,250 cP.

The PP spunbond substrate laminated on one side of the cast film, preferably the outer layer, is a 30 gsm “Ink Blue Polypropylene” “Grade 25224” manufactured by Ahlstrom Corp., Windsor Locks, Connecticut. The product is treated with a surfactant and an antistatic to make it absorbent and to provide good static decay properties. The basis weight was determined according to ASTM D646-96. Other properties were a thickness at 0.63 psi – 12 mils (ASTM D5729-97); grab tensile strength, MD – 25 lbs, CD – 14 lbs (ASTM D5034-95); elongation, MD –60%, CD – 80% (ASTM D5034-95); and trapezoidal tear, MD –2.4 lb, CD – 3.5 lb (ASTM D5733-95). The PP spunbond laminated on the other side of the cast film, preferably the inner layer, was Ahlstrom’s nominal 20 gsm “Ink Blue Propylene” “Grade 25200”. The basis weight is determined according to ASTM D 646-96. Other properties include thickness at 0.63 psi –10 mils (ASTM D5034-95); elongation, MD

–60%, CD –80% (ASTM D5034-95); and trapezoidal tear, MD –2.0 lb, CD – 3.0 lb (ASTM D5733-95).

**Example 2:**

As a comparative example, a three layer extruded film with each layer (A-A-A) comprising only Chevron's PE 1017 low-density polyethylene was laminated to the same PP spunbonds as in Example 1 with similar processing conditions. Each side of the cast film was laminated to a PP spunbond substrate using Fuller NW 1023 hot melt adhesive as in the previous example.

**Example 3:**

A three layer (A-B-A) co-extruded film was produced. Core layer (B) was made from a blend of a 30% LLDPE (MarFlex™ PE 7325) and 70% LDPE (PE 1017). The properties of MarFlex™ PE 7325 resin, a hexane copolymer and commercially available from Chevron Philips Chemical Company, The Woodlands, Texas are a melt index of 3.5 gms/10 min (ASTM D1238E) and a density of 0.9235 gm/cc (ASTM D1505). PE 1017 resin is described in Example 1 above. The blending was done in-line by using gravimetric feeders and the blend, along with other additives such as pigments, then fed into the extruder B. The outer layers (A) were made using a polypropylene resin Marlex EX01D resin as in Example 1.

To produce the co-extruded film, Marlex EX 01D resin was fed from extruder A, while the blend of PE 7235 and LDPE 1017 resins was fed from extruder B. The two resin stocks were then fed to a three layer feed-block and an ABA selector plug to

produce the desired ABA co-extruded film. Extruded ratio used in this particular example was 12.5-75-12.5 by weight.

Suitable equipment to produce co-ex films is available from Cloeren Incorporated, Texas, USA. Extruder A is a 5 zone extruder with a temperature profile of from about 400 °F in the 1<sup>st</sup> zone increasing to about 555 °F in the 5<sup>th</sup> zone. Extruder B is a 6 zone extruder with temperature profile of about 400 °F in the 1<sup>st</sup> zone increasing to about 555 °F in the 6<sup>th</sup> zone. The dies was maintained at approximately 555 °F.

Each side of the cast film was laminated to a PP spunbond substrate using a hot melt adhesive Fuller HL 1713XZP, which is commercially available from H.B. Fuller Company, St. Paul, Minnesota. This hot melt adhesive has the following physical properties: 180 degree peel (60 sec/75F, 1MIL) – 6.7 lbs/iinch, molten Gardner color – 3; Mettler Soft. Point (ASTM D3461) – 185 °F; viscosity at 250 °F – 9,250 cP (mPa.s); viscosity at 275 °F – 4,700 (mPa.s); viscosity at 300 °F – 3,100 cP (mPa.s); viscosity at 325 °F – 1,575 cP (mPa.s) and viscosity at 350 °F – 1,050 cP (mPa.s). The SAFT (500 gms/in<sup>2</sup> load) is 145 °F. The Fuller HL 1713X ZP adhesive is less viscous than Fuller NW 1023 used in the previous example. Line speed for the lamination process was about 300 fpm.

The PP spunbond substrate laminated on one side of the cast film is a 30 gsm “Ink Blue Polypropylene” designated “Grade 25224” manufactured by Ahlstrom Corp., Windsor Locks, Connecticut. The PP spunbond laminated on the other side of the cast film is Ahlstrom’s 20 gsm “Ink Blue Polypropylene” “Grade 25200.”

Properties of the constituent layers and the results of testing are shown in the following Table.

		Example 1			Example 2			Example 3		
Date of Testing	Units	At time of production	After Aging (approx. 1 month)	After EtO Sterilization	At time of production	After Aging (approx. 1 month)	After EtO Sterilization	At time of production	After Aging (approx. 1 month)	After EtO Sterilization
Spunbond Weight (outer layer)	gsm		30			30			30	
Spunbond Weight (inner layer)	gsm		20			20			20	
Film Weight	gsm		18			18			18	
Adhesive add-on (outer layer)	gsm		3			3			3	
Adhesive add-on (inner layer)	gsm		2			2			2	
Handelometer, MD	grams		89			86			87	
Handelometer, CD	grams		41			47			44	
Dry inner Peel Strength (CD)	g/in	81	137	157	74	138	108	83	178	260
Wet Inner Peel Strength (CD)	g/in	97	193	179	90	37	34	117	267	375
Blood barrier (ASTM F1670)	Pass/fail	pass	pass	Pass	Pass	Pass	pass	Pass	Pass	pass

As can be appreciated in from the Table, the wet peel strength of the Example 1 and 3 nonwoven laminate is significantly higher than the wet peel strength of the Example 2 laminate after aging and EtO sterilization. In order to obtain aging data, the samples were tested from the roll for peel strength. During EtO sterilization the samples were subjected to temperature and humidity as part of the sterilization cycle. It was also noted that the softness and drape of the nonwoven laminates in these examples are better than commercially available nonwoven laminate used for medical applications. Fabric flexibility was measured by a Handelometer, Cat. 211-300 from Thwing Albert Instrument Co, 10960 Dutton Rd., Philadelphia, PA 19154 at a gap of 5 mm. Higher numbers correlate with less flexibility. The peel strength was measured using Ahlstrom

internal test method (No. TM 181) using a Zwick tensile tester (Model z 2., Zwick USA LP (Kennesaw, Atlanta, Georgia) to measure the force required to separate component layers of a laminate. Specimen size was 2" x 6". The layers were separated manually for a distance of 1" and then mounted in the jaws. The crosshead speed is 12"/min. The average force to de-laminate was recorded. Peel strength in g/in was reported by dividing the average force by 2. For wet peel strength measurement the samples were soaked for approximately one minute and then the above procedure repeated.

As can be seen from a consideration of the Table, the nature of the composite is critical in order to obtain the desired wet/dry peel strength and barrier strength, the properties being particularly noticeable after aging. As shown in the Table, a significant difference with respect to the dry and wet peel strengths is achieved dependent upon the nature of the polymers employed and their arrangement in accordance with the invention.

5. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the above-referenced application or any patent issuing thereon.

Date: October 30, 2007

  
RAHUL K. DHARMADHIKARY